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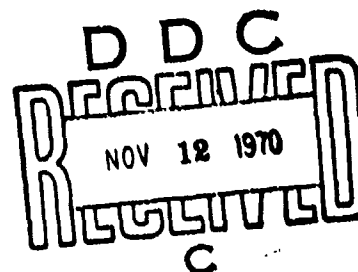
CHANGE IN PROPERTIES DURING AGING OF ALUMINUM ALLOYS

by

I. N. Fridlyander

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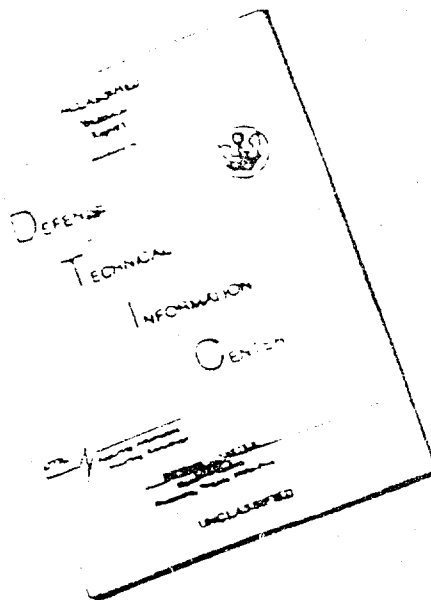


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AUTHOR: I. N. Fridlyander

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An SSSR Institute of Metallurgy - Moscow, 1968,
pp. 90 - 101

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CHANGE IN PROPERTIES DURING AGING OF ALUMINUM ALLOYS

by

I. N. Fridlyander

According to A. A. Bochvar /1/, the type of heat treatment, based on the processes of a dissociation of a state of an alloy fixed by hardening, is called annealing or aging. Annealing or aging can be spontaneous, if the temperature of the space surrounding the hardened alloy is so high that its atoms are capable of migrating inside the lattice. In those cases, when the temperature is too low for spontaneous annealing, the alloy must be heated so that the mobility of the atoms is increased. Such heating of the alloy is called artificial annealing or artificial aging. In relation to the stage of annealing, strengthening annealing at temperatures close to the beginning of the dissociation temperature for a solid solution differs from softening annealing at higher temperatures.

We will designate the following terms to mean: "natural aging" or "cold aging" for the case of aging, without preheating occurring under natural conditions, and "artificial aging" or "hot aging", as that caused by the special heating after hardening.

However, in a number of articles it is shown that, during artificial aging in distinction to natural aging, not only are the processes of dissociation of the supersaturated solution accelerated, which was pointed out by A. Vil'm, but another structure and a different complex of mechanical and physical properties of the alloys appear.

When increasing the duration of the artificial aging or when increasing the aging temperature above the conditions, which lead to maximum hardness, there occurs a lowering of the hardness and creep limit, and elongation increases insignificantly or doesn't change, remaining at a comparatively low level. This stage of the process of dissociation of a supersaturated solid solution is called "overaging".

Silkok, Hill and Hardy /5/, who established the direct connection

between the hardness and structure of the alloys of aluminum with 2.0 and 4.5 percent Cu, have shown that the maximum of the hardness is connected to the presence of the θ'' phase. Hardness is lowered to the extent that the content of the θ' phase is increased. In an alloy with three percent Cu, the maximum of hardness corresponds to a structure, which contains 90 percent θ' and 10 percent θ'' . Consequently, "overaging" meaning a lowering of hardness can occur with the appearance of separations not of the stable, but of the metastable phase.

Many research reports have shown that the dissociation of a solid solution occurs, as a rule, in several stages: the formation of the Guin'ye-Preston zones; the appearance of particles of the metastable phase; and the formation of particles of the stable phase [4,9,11,14-18]. Those changes of properties, which are inherent in natural aging (although they should occur also with heating), are created by the formation of the Guin'ye-Preston zones. Artificial aging and overaging are connected with the appearance of the particles of the metastable phase (and in the case of overaging, possibly, of the stable phase.) The formation of the structure with particles of the stable phase leads to annealing of the hardened alloy. In accordance with this in articles [7 and 8] it was suggested that for the terms natural and artificial aging the terms zonal and phase aging be substituted for the characteristics of the structural changes in the alloy.

Zonal aging does not lead to softening of the alloy for any increase in the duration of the holding time. Phase aging can be hardening or softening (for the overaging stage.) Therefore, it is necessary to differentiate between hardening or softening phase aging and overaging or coagulation (instead of artificial aging or overaging.) For the sake of brevity we can evidently call hardening phase aging or annealing--phase aging, and call softening phase aging or annealing--coagulation during aging.

The transition from the zones to the metastable and then to the stable phases occurs gradually: in the transition period, zones and metastable phases occur, and then metastable and stable phases. Thus, the amount of θ'' in the alloy Al-4%Cu in the aging process at 180° increases from 0 after holding for one hour to predominant values for holding for 100 hours, and is reduced again to 0 after 500 hours of holding. The θ' phase appears after 70 hours of aging and becomes predominant after holding for 500 hours. The change in the properties, for example, the raising of the creep limit and the lowering of elongation, created by the appearance in the structures of metastable phase particles, also occurs in the aging process not by jumps, but gradually with a greater or lesser degree of intensity in relation to the aging temperature.

The transition from zones to phases can be accomplished isothermally when increasing the holding time or when increasing the temperature. For each alloy temperature-time fields can be set up for the zonal, mixed and phase aging and coagulation during aging (fig. 1) If the boundaries of the transitions are put into coordinates of the logarithm of time, then in a number of cases they take the form of straight lines.

According to the degree of the increase, the temperatures of transition from zones to phases accelerate; at sufficiently high temperatures zonal aging can be prolonged for several seconds, at 20°C in alloys of Al-Zn-Mg the transition from zones to phases takes many months or years [12,13.] Below a certain temperature, which is characteristic for each alloy, the transition from zones to phases does not occur, in this case zones exist for an unlimited time. If the appearance of particles of the metastable phases is connected with a sharp rise in the creep limit and an intensive lowering of elongation, then at high temperatures the transition from zones to metastable phases as a result of the return leads to the appearance of extremal points (a maximum and minimum) of hardness properties which are clearly expressed on the curves of the change.

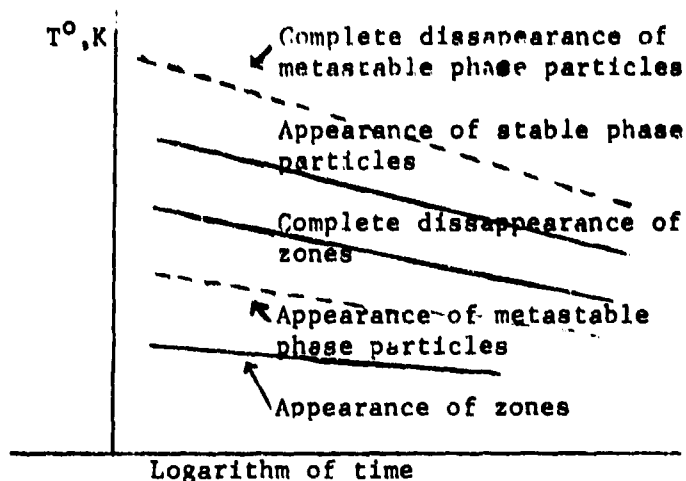


Figure 1. Diagram of the temperature-time fields of transition from zonal to phase aging and annealing.

For the concrete case of the V95 alloy of the Al-Zn-Mg-Cu system (figure 2), aging at 120, 140, 160, 180 and 220°C leads to the appearance of two smooth maximums of the hardness and creep limits. The first maximum appears in the first 5-60 seconds (in relation to the aging temperature.) The hardness in the first maximum in the temperature interval 120-200°C remains unchanged. The hardness in the second maximum reaches the greatest values for the very lowest temperature of its appearance. The creep limit in the second maximum closely approximates the hardness limit. Up to the transition to the second rise of hardness, elongation doesn't change or grows a little and electrical resistance increases. In the second maximum elongation and electrical resistance drop sharply. Aging up to the first maximum of hardness can be characterized as the zonal stage, occurring at high temperatures very rapidly; from the first to the second maximum as phase aging. After the second maximum annealing of the alloy occurs.

At high temperatures the transition takes place so slowly, that the extremal points on the curves of the change of properties are not affected (aging at 20°C) (see figure 2.)

Previous zonal aging affects the actual effect on the subsequent phase aging. In proportion to an increase in the duration of the preliminary natural aging those hardenings in the aging process at high temperature up to reaching

the first maximum of hardness--everything slows down, and then instead of hardening of the alloy there occurs its softening (the return phenomenon.) The softening of the alloy in the process of artificial aging occurs in that case, when the hardness reached during natural aging exceeds the hardness value in the first maximum during artificial aging. Preliminary natural aging noticeably affects also the level of the second hardness maximum, affects the time necessary to reach it, and affects those softenings, occurring after reaching the second maximum for a given condition of artificial aging. The second hardness maximum is lowered in the case of a certain duration of natural aging, and then everything increases, and subsequently everything slows down in the case of further increases in the duration of natural aging.

At insufficient density of the zones (the number of zones per unit of volume) in the case of insufficient length of natural aging, the hardness of the alloy after phase aging seems to be decreased. At larger density of the zones, the hardness characteristics increase; subsequently, during an increase of the duration of previous zonal aging the hardness characteristics of the alloys pass through a minimum, and then exceed the values of those amounts, which correspond to alloys artificially aged immediately after hardening (figure 3.) This is evident for the example not only of alloys of Al-Zn-Mg-Cu, but also for the case of alloys of Al-Mg-Si (figure 4.) Thus, between the zones and the particles of the metastable phases arising later there exists a definite connection; probably, part of the zones allotropically change over into metastable phases or by another method the zones affect the formation of the metastable phases; in relation to the density of the zones the dispersion of the particles of the metastable phases changes,^{/13/} and possibly, also their crystal structures and orientation in relation to the matrix. A similar dispersion of particles of the metastable phases is reached after preliminary zonal aging at substantially higher temperature (and correspondingly for shorter time periods,) than in the case of phase aging immediately after hardening. Consequently, thermal stability of the alloys in the stage of phase aging increases when using previous zonal aging. The effect of zonal aging on the subsequent phase aging clearly appears during gradual aging. In the first stage an accelerated zonal aging occurs, and in the second stage accelerated phase aging at high temperatures: their combination ensures high dispersion of the particles of the metastable phases and high hardness characteristics when shortening the overall aging time.

There occur very definite regularities of change in the properties for all aging aluminum alloys outside the relationship to the special features of the phase and chemical compositions for different stages of aging. In zonal aging the electrical resistance in the process of aging increases; in phase aging and coagulation during aging it decreases.

For zonal aging there is a characteristically lowered value for the creep limit, a considerable discontinuity between the creep limit and the hardness limit, the relation $\sigma_{0.2}$ and σ_{H_p} , as a rule, is less than

0.6 and 0.7; elongation is great--usually not less than 12-15 percent. The high values of elongation are achieved basically on account of the increased uniform elongation, local elongation in the neck zone and narrowing of the cross section do not substantially differ from these characteristics in the phase aging stage (table 1.) The lowered creep limit of the alloys in the

Table 1. Uniform and local (in the discontinuity zone) elongation of alloy V92 on a base of 5 and 2 mm.

| Aging conditions | $l_0 = 5 \text{ mm}$ | | $l_0 = 2 \text{ mm}$ | |
|---------------------------------|--|---------------------|--|---------------------|
| | delta, % at the disloc- ation site | delta, % uniform | delta, % at the dis- location site | delta, % uniform |
| 20°C, 8 days | 35.7 | 13.0 | 46.4 | 13.1 |
| 100°, 96 hours | 22.3 | 8.0 | 35.5 | 7.5 |
| 60°, 24 hours + 195° 2 hours | 29.0 | 5.8 | 44.5 | 6.7 |

zone stage of aging is explained by the fact that the zones do not affect the existing larger resistance to the motion of dislocations, than the matrix; the dislocations cross the zone, and do not bend around them./11//

During phase aging the creep limit closely approximates the hardness limit ($\sigma_{0.2}/\sigma_b = 0.8$ to 0.95), elongation is lowered by 5 to 7 percent on account of the lowering of uniform elongation, hardness reaches maximum values. The high values for the creep limit are created by considerably larger resistance to the motion of the dislocations of the particles of the metastable phases (in comparison to the zonal phases.) The dislocations bend around the particles of the metastable phases, forming multiple dislocation loops around them/11/.

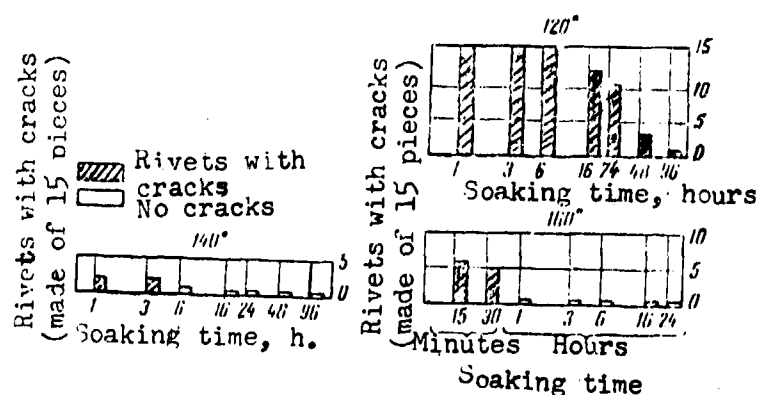


Figure 5. The effect of temperature and aging time on the unriveting of riveted wire made of V 94 alloy. The deposition of the wire with a height of 1.4 d /20/.

In the coagulation process during aging ("overaging"), the particles of the metastable phases become enlarged, the distance between them increases, the motion of the dislocations bending around the particles is facilitated in correspondence to the Orowan criterion [11], and the creep limit decreases somewhat. Uniform elongation continues to remain low, but local elongation and narrowing of the cross section grows and, as a result, the overall elongation increases slightly. The capacity for considerable plastic local deformation, for example, the fastening of rivets, actually improves (figure 5).

The regularities of the changes of σ_b ; $\sigma_{0.2}$; δ ; and Ψ described above appear to be general for all aging aluminum alloys. For all of them there are temperatures and soakings during aging which give high elongation and a low $\sigma_{0.2}/\sigma_b$ ratio, which corresponds to zonal aging; then at certain temperatures and soakings a sharp rise in the creep limit and a lowering of elongation sets in; in this case electrical resistance is also lowered; all this characterizes the transition to phase aging. At even higher temperatures and longer soakings $\sigma_{0.2}$ begins to drop; Ψ which remains comparatively inert all this time, begins to increase and correspondingly elongation increases somewhat and there is a sharp improvement in the technical properties, characterized by considerable plastic deformation (for example, the ability of wire to be unriveted.) Although these regularities are general, the temperature and transition for different alloys differ greatly. For example, for alloys of Al-Zn-Mg the transition from zonal to phase aging sets in at 20-70°, for alloy D16--in the region of 100°, and for alloys of Al-Mg-Si at even higher temperatures. For certain alloys artificial aging conditions occur in the zonal aging range.

It is interesting to note, that the $\sigma_{0.2}/\sigma_b$ ratio is larger and the elongation is less for tempered alloys (in comparison to annealed alloys), which is connected with the conditions of the passing of a dislocation in a structure without separations and to particles of the stable phases, close in the first case to the structure with zones, and in the second case with particles of the metastable phases. At the same time narrowing is greater in tempered alloys.

The impact viscosity and specific operation of breakdown with previously applied cracks has a high value during aging, they are sharply lower as a result of phase aging -- alloys D16 and V92Ts (table 2) and again increase in the process of coagulation during aging for alloy V93 (table 2, figure 6.) Prolonged hardness, fatigue and static endurance change during zonal and phase aging slightly (tables 3 and 4), probably because in the process of more prolonged testing there occurs an additional local dissociation of the supersaturated solid solution and a transition from zonal to phase aging.

Annealing is accompanied by a certain increase in static endurance.

Very definite regularities occur in the relationship of the tendency of alloys to delayed destruction and corrosion under pressure. If for an alloy there exists a tendency for this type of damage (for example, for pure aluminum or low-alloy alloys similar phenomena are not observed), then only one appears at definite conditions of aging, which correspond to a sharp rise in the creep limit and lowering of elongation. For a low creep limit or after the creep limit has reached a maximum, or it noticeably begins to get lower, destructions of the corrosion cracking type do not occur. This is seen for the example of alloys D16/21/, AK4-1, 3D17 and D20 (table 5) and alloy ATSMU (figure 7), but can be set up also for all the other aging alloys, if they, in principle, tend to corrosion cracking. Thus, dangerous conditions in relation to corrosion cracking of aging can be exposed without conducting corrosion tests according to the kinematic curves of the change of $\sigma_{0.2}$ and δ . A similar interconnection of the curves of the change of $\sigma_{0.2}$, δ and the tendency to corrosion cracking can be interpreted in the following manner.

In the case of zonal aging (small $\sigma_{0.2}$, large elongation), when between production and the matrix there is no separation boundary, all the aging alloys are not subjected to corrosion under pressure. The transition from zonal to phase aging leads, evidently, to the appearance of a continuous chain of highly dispersed separations of particles of the metastable phases; the aging alloys become subjected to corrosion under pressure. During further phase aging and coagulation during aging the particles of the metastable phases become harder, the distance between them increases, the continuity of the chain of separations is broken and the alloys again become unsuceptible to corrosion under pressure.

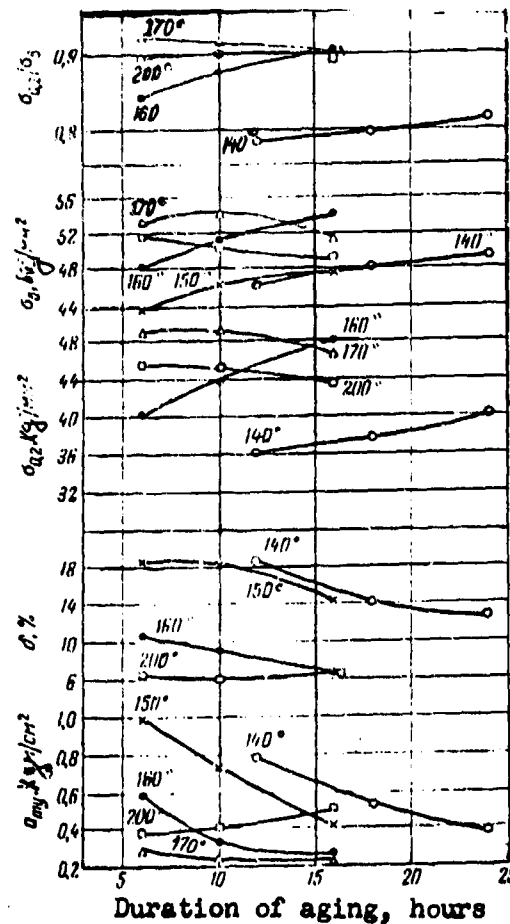


Figure 6. Effect of aging conditions on the mechanical properties of plated sheets of alloy VAD23 (I.N.Fridlyander, Z.N.Archakova, T.K.Zilova)

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Table 2
Impact viscosity of certain aluminum
alloys in relation to aging conditions

| Alloy | Aging | Impact viscosity, kg-m/cm ² |
|-------|-----------------------|---|
| D16 | Natural(zonal)... | 2,0 |
| | Artificial (phase) | |
| | 190°, 6-9 hours | 1,0 |
| V93 | 120°, 3 h + 165°, 4 h | 0,52 |
| | 120°, 3 h + 170°, 5 h | 0,82 |
| | 120°, 3 h + 180°, 5 h | 1,01 |
| V92ts | 20°, 3 months | 2,1 |
| | 80°, 96 hours | 1,5 |

Table 3
Static endurance of certain aluminum alloys
in relation to the aging conditions ($\sigma = 0.1\sigma_b$)

| Alloy | Aging conditions | Static endurance, (number of cycles) |
|--|-------------------------|---|
| V92ts, sheet | 20°, 8 months | (7-10) · 10 ³ |
| | 80°, 96 hours | (8-10) · 10 ³ |
| | 20°, 1 month | (5-8) · 10 ³ |
| 01915, (Al-Zn-Mg) Profile of V93, forging in the height direction | 100°, 24 h + 150°, 10 h | (4-6) · 10 ³ |
| | 120°, 3 h + 165°, 4 h | 3 · 10 ³ |
| | 120°, 3 h + 175°, 5 h | 4,2 · 10 ³ |
| | 120°, 3 h + 190°, 5 h | 6,3 · 10 ³ |

Table 4
Fatigue of certain aluminum alloys at
different aging conditions
Based on 2 x 10⁶ cycles

| Alloy | Aging conditions | fatigue limit, kg/mm ² | |
|-------|-------------------|-----------------------------------|----------------------|
| | | smooth specimens | specimens with holes |
| D16 | natural aging | 10 | 7 |
| | artificial aging, | | |
| | 190°, 6 h | 10 | 8 |
| V92ts | 20°, 3 months | 13 | — |
| | 60°, 24 h + 195°— | | |
| | 2 h | 12,8 | — |

Table 5

Relationship of mechanical properties and corrosion resistance of alloys AKh-1, VD17 and D20 to the aging conditions

| Alloy | Aging conditions | Mechanical properties | | | Time up to destruction of specimens, in days |
|-------|-----------------------------|-------------------------------------|-------------------------------------|--------------|--|
| | | $\sigma_{0.2}$, kg/mm ² | $\sigma_{0.5}$, kg/mm ² | δ , % | |
| AKh-1 | 170°, 10 h . . . | 42 | 33 | 10 | 8 |
| | 185°, 16 h . . . | 42,5 | 37,8 | 14 | 8 |
| | 185°, 24 h . . . | 43 | 40 | 10 | >100 |
| | 195°, 16 h . . . | 44 | 30 | 13 | >100 |
| | 200°, 5 h . . . | 42,5 | 37 | 14 | 11 |
| | 200°, 10 h . . . | 43,8 | 30 | 13 | >100 |
| | natural aging, 7 days . . . | 41 | 20 | 20 | >40 |
| VD17 | 170°, 10 h . . . | 44 | 31 | 21 | 7 |
| | 190°, 10 h . . . | 44 | 34 | 18 | 12 |
| | 200°, 10 h . . . | 43 | 41 | 14 | >100 |
| | 200°, 24 h . . . | 42 | 37 | 10 | >100 |
| | natural aging, 5 days . . . | 44 | 20 | 12 | >100 |
| D20 | 170°, 16 h . . . | 42 | 28 | 14 | 6 |
| | 200°, 10 h . . . | 39 | 25 | 13 | 3 |
| | 200°, 16 h . . . | 38 | 25 | 13 | >40 |

Notes. 1. Corrosion resistance was determined by variable submersion in 3% NaCl; 2. $\sigma_{0.2}$, $\sigma_{0.5}$.

Table 6

Mechanical properties and corrosion resistance (time to destruction of specimens) of sheets made of alloy V92ts after different aging conditions and additional low-temperature heating

| Additional heating | 20°, 6 months | | | | 20°, 24 h + 100°, 24 h | | | |
|-----------------------|-------------------------------------|-------------------------------------|--------------|---------------------------------|-------------------------------------|-------------------------------------|--------------|--|
| | $\sigma_{0.2}$, kg/mm ² | $\sigma_{0.5}$, kg/mm ² | δ , % | time up to destruction, in days | $\sigma_{0.2}$, kg/mm ² | $\sigma_{0.5}$, kg/mm ² | δ , % | time up to destruction of specimens, in months |
| without heating . . . | 45,0 | 20,3 | 10,0 | >6 mo. | 48,2 | 34,4 | 11,7 | >6 |
| 70°, 500 hours . . . | 48,3 | 34,0 | 14,0 | ~14 | 40,6 | 30,2 | 11,0 | >6 |
| 70°, 1000 " . . . | 40,5 | 30,7 | 14,0 | ~18 | 47,3 | 37,0 | 10,4 | >6 |
| 70°, 3000 " . . . | 52,4 | 40,1 | 12,0 | — | 40,0 | 30,2 | 10,8 | >6 |

Alloys also differ by the tendency to corrosion under pressure and intercrystalline corrosion in relation to the temperature-time interval of the transition from zonal to phase aging. Those alloys do not tend to corrosion under pressure, for which aging at room temperature, at operational temperature (aerodynamic or nozzle heating), technical heating or the conditions used for artificial aging lead to the appearance of the structure of either zonal dissociation or well developed phase aging. On the contrary, the transition to phase aging is characterized by maximum sensitivity to corrosion cracking.

It is necessary to note that zonal structure can be subjected to further dissociation under the influence of applied pressures (during operations or in tests), of nozzle heating or very prolonged stay at room temperature. In order to evaluate this capacity of the alloys in /8, 22/ a method was proposed for prolonged holding of specimens (up to 3000 hours) at temperatures of 50 and 70°, which allowed the appearance of the possible tendency to corrosion cracking (table 6.) On the other hand, heating at higher temperatures of the alloy, located in the phase aging stage, improves its resistance to corrosion cracking (table 7.)

In principle for each aging alloy it is advantageous to have three aging conditions, which correspond to zonal aging, phase aging and coagulation

during aging. In zonal aging maximum plasticity is observed at sufficient strength and at average creep limit, but also with high sensitivity to possible subsequent heating. Phase aging gives maximum strength and creep limit, but lowered elongation -- danger of corrosion cracking (in the initial stage of aging) and high sensitivity to cracks. Coagulation during aging ensures high corrosion resistance, a high creep limit, weak sensitivity to additional heating (operational and test), increased plasticity, especially test, however all the relative elongations continues to remain low, and the sensitivity to cracks remains high.

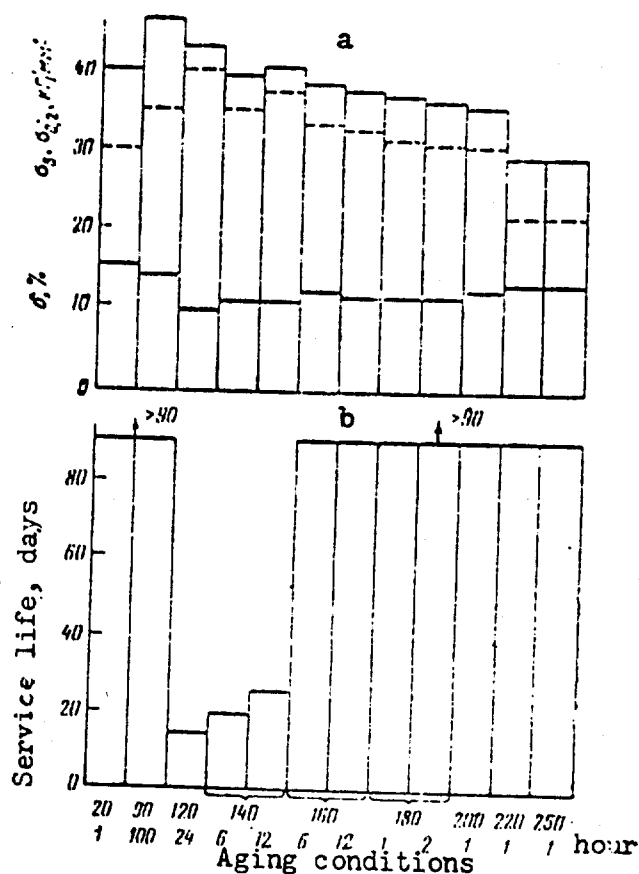


Figure 7. The effect of aging conditions ($^{\circ}\text{C}, \text{hrs}$) on the mechanical properties and corrosion resistance under pressure of alloy ATsMU (Fridlyander, Kuznetsova, Davydova, Batrakova and Surova)

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Table 7
Properties of alloy V93 after various aging conditions

| Properties* | Aging conditions | |
|--|--------------------------|--------------------------------------|
| | 120°. 3 h + 165°. 4 h | 120°. 3 h + 170°. 4 h |
| $\sigma_{0.2}$, kg/mm ² | 50,1 | 32,0 |
| σ_b , kg/mm ² | 52,8 | 41,4 |
| δ , % | 7,8 | 10,3 |
| Duration of life of specimens, in days | 28 | more than 120, tests discontinued |

* Specimens were cut out of die stamps in the height direction

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| 13. ABSTRACT <p>According to A. A. Bockvar (1), the type of heat treatment based on the processes of a dissociation of a state of an alloy fixed by hardening, is called annealing or aging.</p> <p>During artificial aging in distinction to natural aging not only are the processes of dissociation of the supersaturated solution accelerated but another structure and a different complex of mechanical and physical properties of the alloys appear.</p> <p>When increasing the duration of the artificial aging or when increasing the aging temperature above the conditions, which lead to maximum hardness there occurs a lowering of the hardness and a creep limit, and elongation increases insignificantly or doesn't change remaining at a comparatively low level.</p> | | |

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